NASw-101

UNPUBLISHED PRELIMINARY DATA

RESEARCH REPORT

N65 16257
(ACCESSION NUMBER)

12
(PAGES)

CR 60511
(NASA CR OR TMX OR AD NUMBER)

(THRU)

1
(CODE)

17
(CATEGORY)

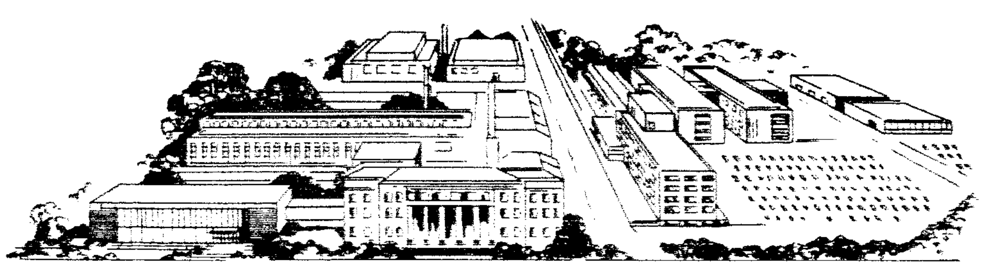
FACILITY FORM 602

GPO PRICE $ ______

OTS PRICE(S) $ ______

Hard copy (HC) 1.00

Microfiche (MF) .50



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NINETEENTH QUARTERLY PROGRESS REPORT

on

INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

January 4, 1965

by

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INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

INTRODUCTION

Experimental work during the present report period has been proceeding in three main directions, with the object of investigating the effect of rhenium in modifying the properties of the brittle refractory metals. Progress in each section will be described separately.

SECTION I – EFFECT OF RHENIUM ON NITROGEN PRECIPITATION IN CHROMIUM

The report prior to this one described a completed segment of research designed to investigate the effect of rhenium on the fracture mode of tungsten. The object of this work was, in part, to determine whether rhenium additions of 3-5 at. % modified the grain-boundary precipitate morphology in such a way as to lead to enhanced ductility. No evidence was found for this effect. In the present report period, research is being completed to investigate whether higher rhenium additions cause any change in the grain-boundary precipitates in chromium.

The work was carried out on Cr-35Re wires 39 mils in diameter that had been loaded with 170 ppm of nitrogen at high temperature and then quenched to retain the nitrogen in solution down to room temperature. A sample of this material was tested in the as-quenched condition, and another in the quenched and aged condition. The specimens were tested at -196 C in a 3-point bend-test apparatus. Strain was applied by an Instron testing machine at a crosshead speed of 0.02 in./min, and the load was recorded autographically. The load-time curves are presented in Figure 1, from which it can be seen that the quenched specimen twinned prior to failure at a load of 26 pounds, while the aged specimen failed at 52 pounds' load with no evidence of prior deformation.

Metallography and electron-replica fractography were performed on the fractured specimens in order to investigate the effect of the different treatments on the fracture appearance. Figure 2 shows the fracture surface in the quenched specimens, which consists of exposed grain-boundaries crossed by twins but relatively clean from precipitate. The aged specimen, shown in Figure 3, also failed at the grain boundaries which, in this case, are almost completely covered by a film of precipitate with occasional twins. The metallographic cross section in Figure 3b emphasizes the extent of nitride precipitation at the grain boundaries, and the higher magnification electron replica fractograph in Figure 4 shows the morphology of the precipitate.

In order to determine the nature of this precipitate, techniques were developed to extract it from the grain boundaries. Initial attempts were made to extract the precipitates from the exposed fracture surface by direct carbon deposition followed by

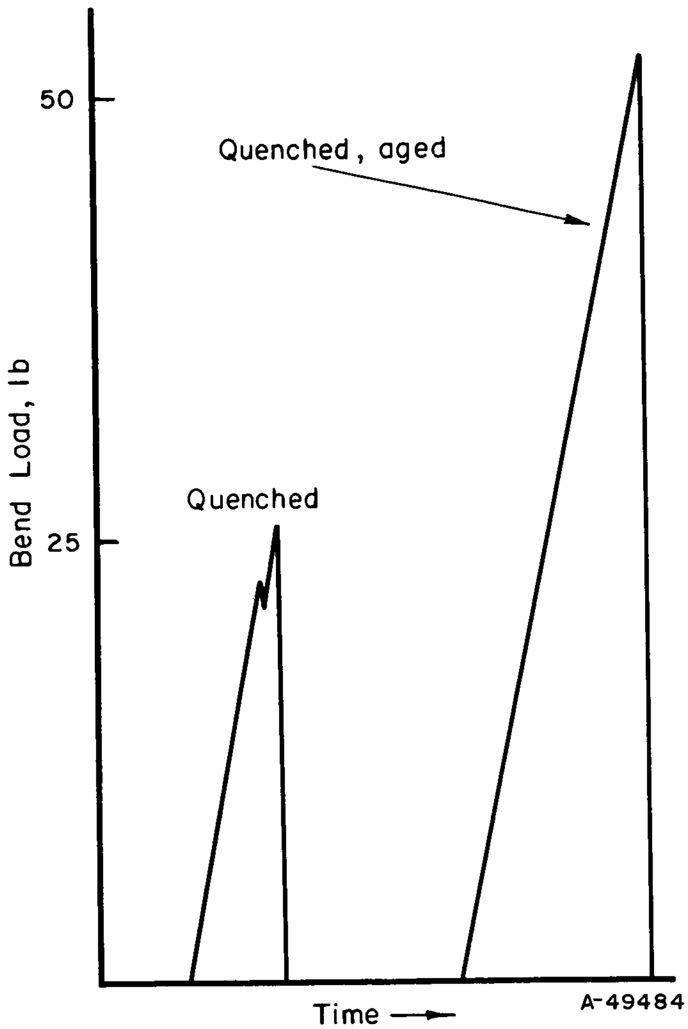


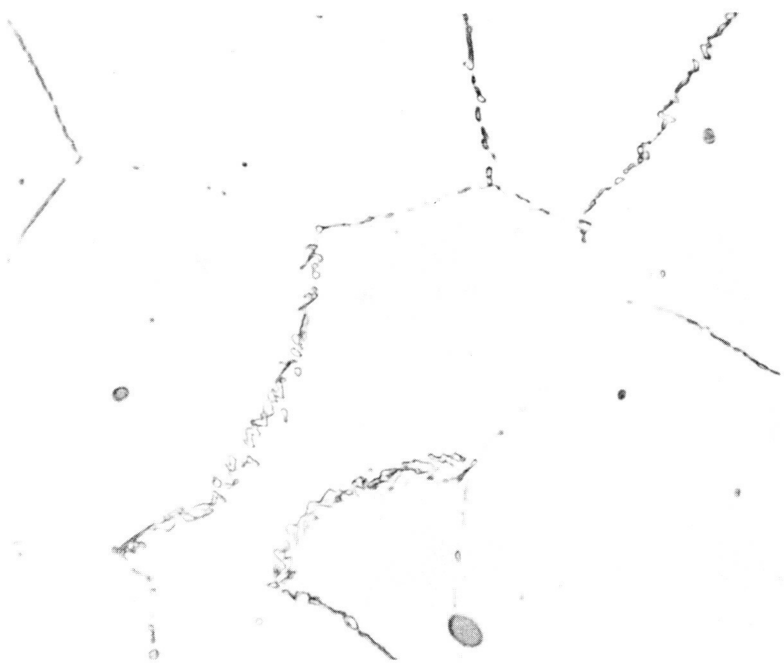
FIGURE 1. LOAD-TIME RECORDS OF BEND TESTS ON N_2 CONTAMINATED WIRES OF Cr-35Re.

1350X E1140A

FIGURE 2. ELECTRON REPLICA FRACTOGRAPH OF THE GRAIN BOUNDARIES EXPOSED BY FRACTURE OF THE QUENCHED Cr-35Re WIRE AT -196 C

1350X E1134C

a. Electron Replica Fractograph of the Grain Boundaries Exposed by Fracture of the Aged Cr-35Re Wire at -196 C



1500X 14979

b. Metallographic Section of Specimen in Figure 3a

FIGURE 3. GRAIN BOUNDARY PRECIPITATES IN AGED Cr-35Re WIRE

5500X E1145A

FIGURE 4. ELECTRON REPLICA FRACTOGRAPH SHOWING THE MORPHOLOGY OF THE GRAIN-BOUNDARY PRECIPITATE

electrolytic dissolution. No success was achieved by this means, however, and eventually precipitates were extracted from heavily overetched grain boundaries by pressing softened acetate sheet onto the etched surface. After these replicas had dried in position, they were stripped from the surface and brought with them specimens of precipitate which they had mechanically extracted. The replica containing the precipitates was then shadowed with carbon, after which the plastic was dissolved. The carbon replica was examined in the electron microscope, and the electron-diffraction patterns were taken of regions containing precipitate. Comparison of the diffraction rings with standard tables showed good agreement between the lines observed and the strongest lines of Cr_2N.

Thus, with the presence of large amounts of rhenium in solid solution, it appears that the nitride phase precipitating is the same as in unalloyed chromium. However, it also appears that large amounts of such precipitate at the boundaries are not necessarily weakening, since, from Figure 1, the aged material sustained a stress prior to failure twice as high as that sustained by the quenched material. An important effect of aging seems to be suppression of twinning. It is hoped to perform more detailed experiments in the coming report period in which the effect of aging will be investigated on the tensile properties of Cr-35Re wires loaded with nitrogen.

SECTION II - EFFECT OF RHENIUM ON THE STRAIN-RATE SENSITIVITY OF CHROMIUM AT ROOM TEMPERATURE

The response of a metal to a rapidly increasing stress (such as exists ahead of a propagating crack) depends on the way dislocations can move in response to an increase in stress. It has been shown for many materials that dislocation velocity obeys an equation of the form

$$v \propto \sigma^m \quad , \tag{1}$$

where v is the dislocation velocity, σ is the stress experienced by the dislocation, and m is a constant for a particular material.

Thus,

$$\log v = m \log \sigma \quad . \tag{2}$$

In order for plastic flow to occur in advance of a moving crack sufficiently rapidly to blunt and arrest the crack, let us assume that dislocations must move at velocity, v'. Other things being equal, this velocity, v' (or rather $\log v'$), can be attained at a stress which from Equation (2) is seen to be inversely proportional to m. Thus, for high values of m, dislocations can attain this velocity, v', at a much lower stress than for low values of m. Since the stress-concentrating effect of a crack falls off approximately inversely with distance ahead of the crack, large values of m permit plastic flow to occur well ahead of the crack, thus maximizing the possibility of the crack stopping.

In order to devise an experimental technique for determining m for a given material, the following reasoning is invoked.

Since plastic flow occurs by the movement of dislocations, the plastic strain rate, $\dot{\epsilon}_p$, can be written as a product of ρ, the number of dislocations moving and v, their average velocity:

$$\dot{\epsilon}_p \propto \rho v \quad . \tag{3}$$

Using Equation (1),

$$\dot{\epsilon}_p \propto \rho \sigma^m \quad . \tag{4}$$

If it is assumed that the number of dislocations moving is independent of strain rate, then Equation (4) can be written

$$\log \left(\frac{\dot{\epsilon}'_p}{\dot{\epsilon}''_p} \right) = m \log \left(\frac{\sigma'}{\sigma''} \right) \quad , \tag{5}$$

where σ' is the flow stress at strain rate, $\dot{\epsilon}'_p$ and σ'' is the flow stress at $\dot{\epsilon}''_p$.

It has been experimentally determined that the yield stress or flow stress is related to the strain rate by an equation very similar to this, that is,

$$\log \frac{\sigma'}{\sigma''} = n \log \frac{\dot{\epsilon}'_p}{\dot{\epsilon}''_p} \quad . \tag{6}$$

Comparing Equations (5) and (6), it can be seen that $m = 1/n$. Thus, a highly strain-rate-sensitive material is characterized by a large value of n and a small value of m.

The preceding argument shows that, other things being equal, a material which is very strain-rate sensitive should be more susceptible to cleavage propagation than one which is insensitive to strain rate.

In an earlier report, it was shown that for strain-rate cycling by a factor of 10, Cr-35Re was an order of magnitude less sensitive to strain rate than was chromium. The object of this portion of the present work is to document the effect of rhenium on the room-temperature strain-rate sensitivity of chromium over a range of strain rates.

To this end, compression samples of Cr and Cr-15Re have been tested at room temperature at strain rates from 2×10^{-3} up to 1 per minute. The proportional limit has been chosen as the relevant flow-stress parameter, and has been measured from stress-strain curves obtained at the different rates. Table 1 and Figure 5 show the results obtained to date. Samples of Cr-35Re are prepared in order to extend the range of rhenium compositions to the most ductile alloy and are to be tested soon. In addition, it is hoped to extend the range of available strain rates by another factor of 100 through the use of a newly acquired high-strain-rate testing machine.

TABLE 1. EFFECT OF STRAIN RATE ON PROPORTIONAL LIMIT OF CHROMIUM AND CHROMIUM-15 RHENIUM

Composition	Strain Rate per Minute	Proportional Limit, 1000 psi
Cr	0.002	14
Cr-15Re	0.002	52.4
Cr	0.01	16
Cr	0.1	25
Cr-15Re	0.1	57.1
Cr-15Re	0.2	61.0
Cr-15Re	1.0	74.5
Cr	1.2	41.5
Cr	2.0	65.0
Cr-15Re	2.0	94.5

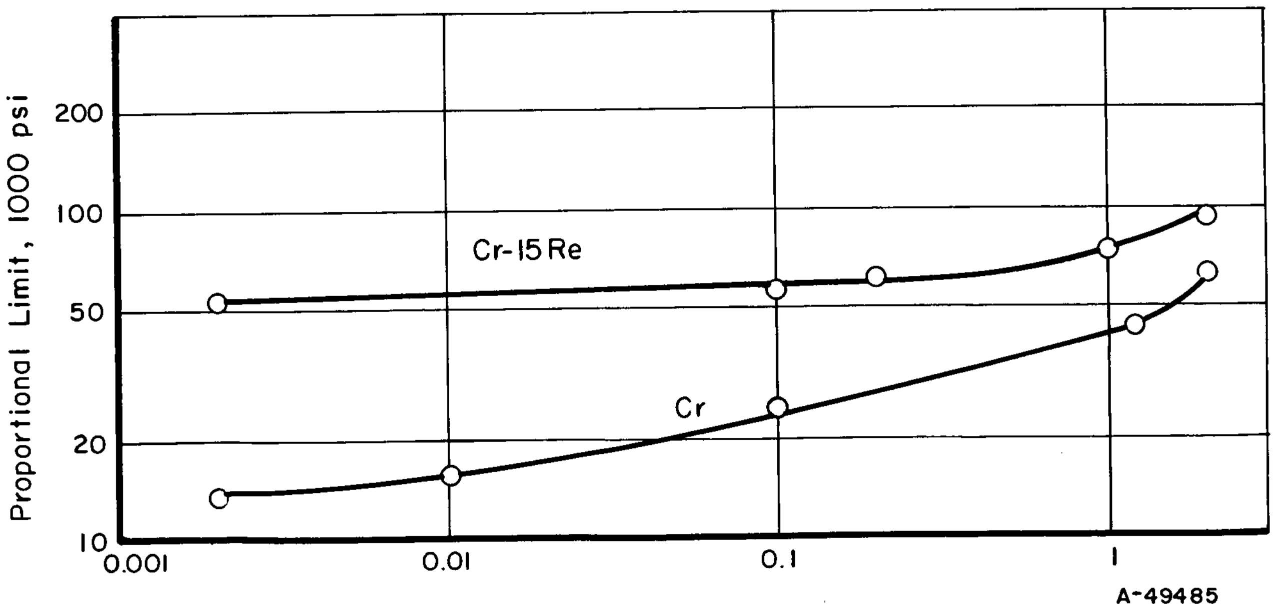


FIGURE 5. EFFECT OF STRAIN RATE ON PROPORTIONAL LIMIT OF Cr AND Cr-15Re

Data obtained in compression at room temperature.

SECTION III - EFFECT OF RHENIUM ON THE SLIP PLANES IN MOLYBDENUM

It may well be that the ductilizing effect of rhenium in the Group VI-A refractory metals is due to a modification of the active slip systems. In order to investigate this possibility, work described in the Seventeenth Quarterly Progress Report was initiated and is still progressing. Insufficient progress has been made at this time, however, to warrant a further presentation, but experiments are still in progress to determine the slip systems active in single crystals of Mo-35Re as a function of temperature and orientation.

AG:cm